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Characterization of amorphous carbon in terms of their turbostratic ordering

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Abstract . This paper presents results of XRD, I R. and other physical studies of amorphous carbon (a-C) prepared by the action of concentrated H_2SO_4 on three carbohydrates *viz* glucose, sucrose, and starch. Reduced radial distribution function (RDF) study reveals that graphitic order (Turbostratic ordering) is highest in sucrose carbon followed by starch and glucose carbon. Starch and glucose carbon yield almost similar reduced RDF pattern and hence it is inferred that starch may break down into glucose moiety before carbonization. This fact is also corroborated by I R and Electrical Conductivity studies. I R study suggests that along with three-fold symmetry (sp^2), certain amount of four fold symmetry (sp^3) is also present, particularly in glucose and starch carbon This study reveals that dehydration products of carbohydrates, are very akin to hydrogenated amorphous carbon (a-C,H) prepared by D C magnetron glow discharge of acetylene

Keywords Amorphous carbon, turbostratic arrangement, characterization

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. Introduction

The structure of a-C has not yet been solved with any certainty, particularly those obtained by dehydration of carbohydrates. It is believed that various forms of a-C has similar short-range order with differences, which depends on starting material, on the method of preparation and on the heat treatment temperatures. No RDF studies on dehydration products of carbohydrates were ever done to understand the carbonization process Several theoretical models of a-C were suggested [1-5], but none of these can exactly explain RDF pattern obtained from experimental data.

Turbostratic arrangement, which is the characteristic of graphite, is also found to exist in amorphous carbon [6] and in Coal [7] Schoening [7] has shown that in case of turbostratic arrangement a peak is always observed at 43 Å in the reduced RDF, *i.e.* $G(r)$ *versus* r plot. Height of this peak is a measure of the degree of graphitic order, the sample under study has attained In case of sucrose carbon, height of 4.3 Å peak was found to be higher than those of glucose and starch carbon. Glucose and starch carbon yield almost similar reduced RDF. Electrical conductivity study also corroborates these facts. Object of this work is to study graphitic order in dehydration products of carbohydrates and characterize the materials in terms of their infrared (I.R.), electrical conductance and X-ray diffraction (XRD) study.

I.R. is a good probe to ascertain the nature of bonding in a-C and electrical conductance data also give certain idea about graphitic ordering. These studies have been carried out and it is revealed that dehydration products of carbohydrates and very akin to hydrogenated amorphous carbon (a-C:H).

2. Theory

Radial distribution function, which means the probability of a 2nd atom to be found at a distance $r+dr$ from a central reference carbon atom, is given by [8]

$$4\pi r^2\rho = 4\pi r^2\rho_a + \frac{2r}{\pi}\int_0^{K_{max}} Ki(K)\exp(-\alpha K^2)\sin KrdK, \quad (1)$$

where $4\pi r^2\rho$ = RDF,

ρ_a = atomic density,

$i(K) = (I'_{eu}/N - f^2)/f^2$ = Interference function ,

$K = 4\pi\sin\theta/\lambda$,

α = damping factor .

In other words

$$\text{RDF} = 4\pi r^2 \rho_a + rG(r), \tag{2}$$

where $$G(r) = \left(\frac{2}{\pi}\right)\int_0^{K_{max}} Ki(K)\exp(-\alpha K^2)\sin KrdK. \tag{3}$$

According to Schoening [7], a plot of $G(r)$ *vs*, r will always give a peak at 4.3 Å, in case turbostratic arrangement is present.

Now, this integral in eq. (3) should converge at $K = K_{max}$. and height of the 4.3 Å peak is a measure of graphitic order a sample has attained.

3. Experimental

XRD studies :

Three carbohydrates *viz* glucose, sucrose and starch were treated with concentrated H_2SO_4 and kept on water bath for 1 hr. Charred products were thoroughly washed with distilled water and air-dried. Their powder XRD patterns were recorded in a Seifert diffractometer with steps of $\theta = 0.02$ deg, scanning rate = ½ deg/min, using CuKα radiation. Counts varied from 40,000 at the most intense part of the diffractogram to 400 in the high angle side. Intensities thus recorded were directly stored in a Floppy in form of a ASCI file and were used to calculate RDF and reduced RDF studies using the program SCATFAC developed by the present author after appropriate corrections. Integration was carried out upto $K_{max} = 7.88$ Å with $\alpha = 0.5 \times 10^{-2}$. It should be mentioned in this context that for CuKα, integration in eq. (3) can not converge for $r = 1$, $r = 2$ and $r = 3$. The corresponding $\sin K_{max}r/K_{max}r$ values is 0.12, 0.33 and 0.042. But for $r = 4$, $\sin K_{max}r/K_{max}r = 3.29 \times 10^{-3}$. In other words, integral in eq. (3) converges with $K_{max}r = 7.88$ and $r = 4$ Angstrom. Therefore, RDF and reduced RDF data beyond $r = 4$ may be considered to be quite reliable and precise. We can safely make use of 4.3Å peak as a measure of turbostratic ordering. In each case, a peak was found at (4.3 ± 0.01) Angstrom.

Reduced RDF of these three a-Cs and that of Phillips India Ltd's amorphous carbon were shown in Figure 1. Conductivity data, density and peak height of 4.3 Å in reciprocal space were reported in Table 1.

Table 1. Schoening's g factor, electrical conductivity and density of three a-carbons *viz* glucose, sucrose and starch

a-C Type	Height of 4.3Å peak	Electrical conductivity cm^{-1} ohm^{-1}	Density gms/C.C
Glucose carbon	0.34	3.2×10^{-1}	1.42
Sucrose carbon	0.51	5.7×10^{-1}	1.495
Starch carbon	0.37	3.6×10^{-1}	1.484

I.R. Studies :

I.R. spectra (Figure 2) recorded in FTIR model no Magna IR750 of Nicolet (USA) strongly support what has been shown

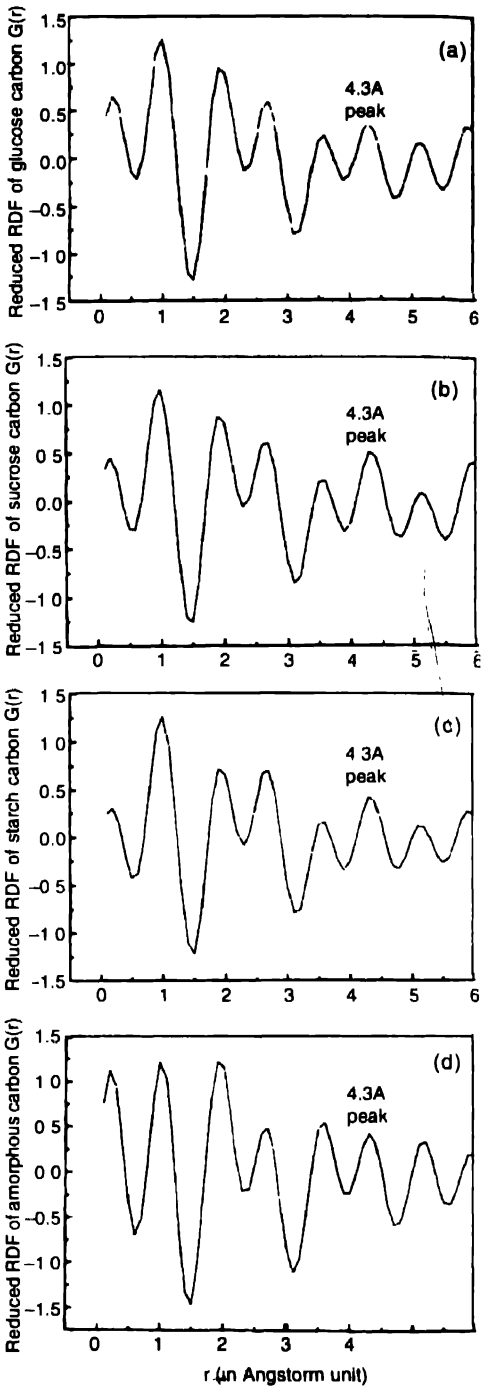

Figure 1. Reduced radial distribution function of (a) glucose carbon (b) sucrose carbon (c) starch carbon and (d) amorphous carbon bought from Phillips India Ltd. Height of the 4.3Å peak is the measure of graphitic order.

by reduced RDF *i.e.* degree of graphitic order is the highest in sucrose carbon followed by starch and glucose carbon, because 1622 cm^{-1} peak which is characteristic of $C = C$ (sp^2 olefinic) strech, is the strongest in sucrose carbon followed by starch and glucose carbon. I.R. spectra of glucose carbon is almost

identical with that of starch carbon, again suggesting that starch may first break down into glucose moiety before carbonization. Presence of four fold symmetry (sp^3) is also inferred particularly in case of starch and glucose carbon, due to the presence of out, because broad 3432 cm^{-1} band may engulf 3300 cm^{-1} band responsible for ≡ CH group. Table 2 shows peak positions in the spectra along with assignment of vibrational mode responsible for such bands. Dischler *et al* [9] and McKenzie

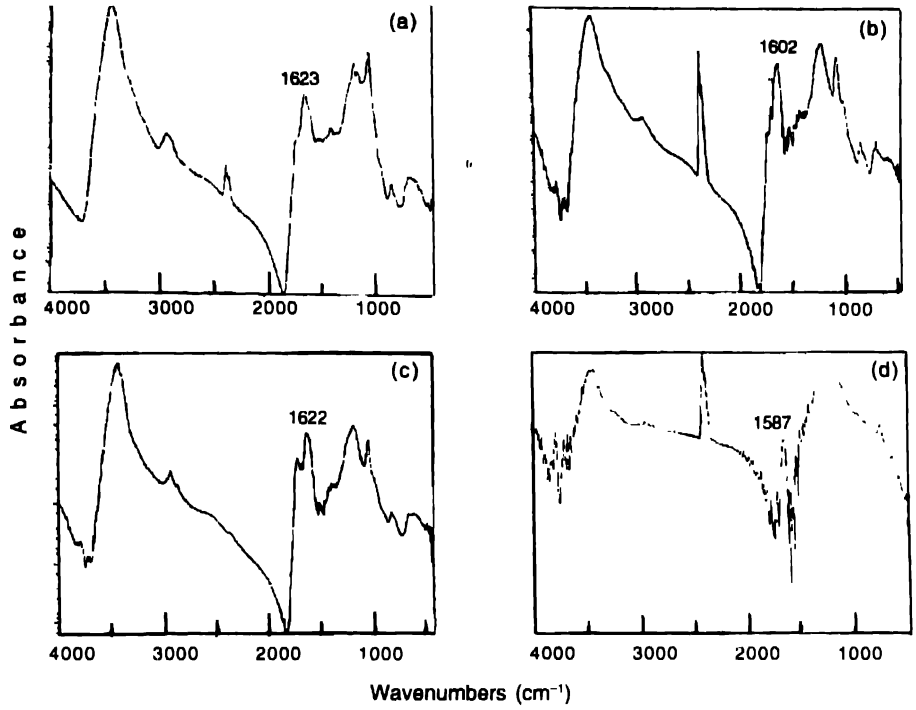

Figure 2. I R spectra of (a) glucose carbon (b) sucrose carbon (c) starch carbon and (d) amorphous carbon bought from Phillips India Ltd peaks characteristic of C = C (1650 cm^{-1}) Sp^2 Olefinic are marked in each figure

1330 cm^{-1} peak characteristic of C – C (sp^3) stretch and 2925 cm^{-1} band responsible for sp^3 CH_2 group in all spectra shown in Figure 2 Presence of *sp* hybridisation can not be totally ruled

Table 2. Infrared absorption bands observed in carbon samples

Band position	Glucose carbon	Sucrose carbon	Starch carbon	Assignment	Theoretically predicted positions
	cm^{-1}	cm^{-1}	cm^{-1}		cm^{-1}
1	3423	3432	3435		
2	2921	2925	2925	Sp^3CH_2	2925
3		1666	11669	> C=O	1600–1700
3'	1623	1602	1622	Sp^2(C=C)	1626
4	1495				
5	1425				
6	1376		1434	Sp^3C-C	1336
7	1315				
8	1161	1187	1162	–COOH	1200–1000
9	1021	1022	1022	≥C–C≤	1022
9'		965			
10	798	784	795		
11	639	647			
12	585		581		

[10] have also observed most of the bands observed by the present author and hence it is deduced that in carbon network *H*-atom is scarcely attached in the periphery.

4. Concluding remark

Characterization of the dehydration products of carbohydrates was done in terms of their graphitic (turbostratic) ordering using XRD and I.R. These are identified as carbon networks containing both three-fold and four-fold symmetry (sp^2 and sp^3) and the former is believed to be dominant. H atoms and functional groups like – COOH > CO are scarcely attached to this network of carbon. A plausible mechanism of carbonisation was provided with, in case of starch carbon. All the carbons produce same I.R. spectra after a gap of six months, which indicate these are chemically stable and can be used as potential electrical insulators since they have high resistivity. As regards their I.R. spectra and density data, these carbons are found to be very akin to hydrogenated amorphous carbon a-C:H, but electrical conductivity is more closer to evaporated a-C (10^{-3} ohm^{-1} cm^{-1}) [11].

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